APPLICATIONS VOID.

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PATENT SPECIFICATION

479,925

Convention Dates: (Germany) { *May 9, 1935. No. 13275/36.* / *July 20, 1935. No. 13276/36.* / *July 20, 1935. No. 13277/36.*

Application Date (in United Kingdom). May 11, 1936.

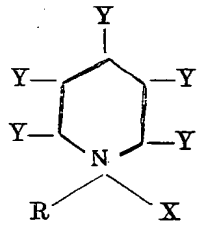
Specification not Accepted

COMPLETE SPECIFICATION

Process for Disinfecting and Preserving

We, CHEMISCHE FABRIK VON HEYDEN AKTIENGESELLSCHAFT, a body corporate organised according to the laws of Germany, of Radebeul - Dresden, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention is based on the discovery that by the addition of a suitable reactive compound R—X to a pyridine compounds having a high bactericidal action can be obtained. The new compounds are accordingly quaternary pyridines of the general formula



In this formula Y stands for hydrogen or any desired residues which may be the same or different, R stands for any desired organic residue and X for any desired organic or inorganic anion.

As suitable compounds there may be named by way of example:

The addition products of alkyl halides and pyridine, for example

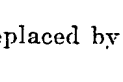
Dodecylpyridinium chloride,
Tetradecylpyridinium chloride,
Cetylpyridinium chloride,
Octadecylpyridinium chloride

and others, or addition products of alkyl halides and homologues of pyridine, for example

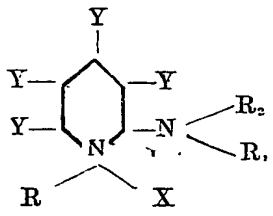
Cetyl-α-picolinium chloride,
Cetyl-γ-*n*-propylpyridinium chloride,
Tetradecyl - γ - isoamylpyridinium chloride.

It is also possible to combine with pyridine or its homologues by addition a compound containing an oxy-group such as ethylene chlorhydrin or α-chlorhydrin and then to esterify the oxy-group, for example with lauric acid, myristic acid, stearic acid or oleic acid. Or an amine such as dodecylamine, tetradecylamine, hexadecylamine, octadecylamine or a 2-alkoxy-5-aminopyridine may be converted into an amide of a halogen carboxylic acid such as chloracetic acid and the amide thus obtained may be combined with a pyridine by addition.

The invention is also based on the discovery that certain α- or γ-pyridonimides and quaternary α- or γ-aminopyridines have a high bactericidal action which renders them suitable for extensive use in cases where the extermination of micro-organisms comes into consideration.

There come into question compounds in which one Y in the foregoing formula has been replaced by the  group

and to which there may probably be attributed, in the form of their salts, the following formulæ:



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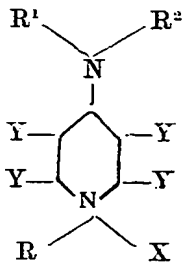
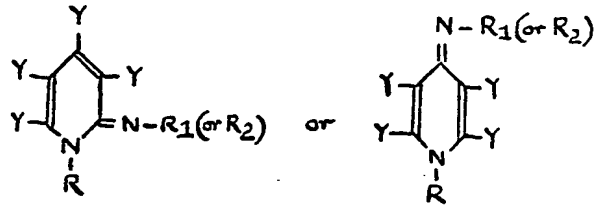


in which X represents any desired organic or inorganic anion, advantageously a halogen atom or a derivative of sulphuric acid, Y represents any desired residues which may be the same or different, R represents any desired organic residue, for example an alkyl group or an aralkyl group, and R_1 and R_2 represent hydrogen or any desired organic residues which may be the same or different. The residues R_1 and R_2 may be linked to the α- or γ-amino nitrogen atom either in the manner of an amine, for example in the case of alkyl, cylcoalkyl, aralkyl, aryl or heterocyclic groups, or in the manner of an amide in the case of radicals of saturated or unsaturated fatty acids or cycloaliphatic, aromatic-aliphatic, aromatic or heterocyclic acids. The residues R, R_1 and R_2 may also be substituted in any desired manner, for instance by halogen atoms, ether or thioether groups, amino groups and so on or their carbon chains may be interrupted by other atoms.

According to the solubility required of the compounds they may be used in the form of their salts of various acids. If required the free bases may be prepared from the salts and may be used as such.

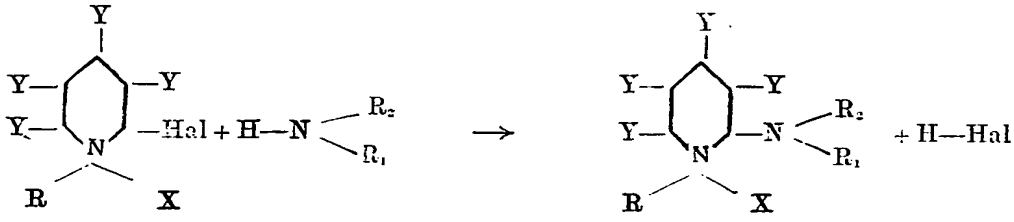
If R_1 and/or R_2 is hydrogen the free bases are of low solubility and pass with loss of water or acid into pyridonimides represented by the following formulæ:



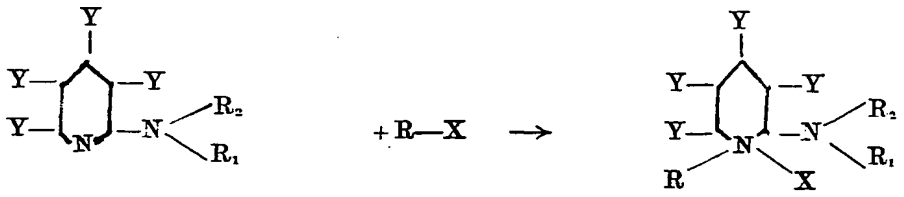
Depending on their solubility these may be used either as such or in the form of their salts with suitable acids.

Compounds of the aforesaid kind are obtainable according to various methods, some of which are known. Without exhausting all possibilities the following reactions may be named:

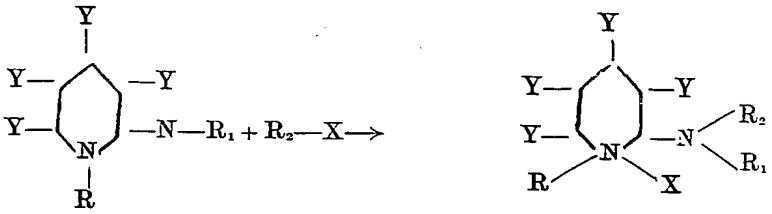
The reaction of a quaternary α- or γ-halogen pyridine (or another suitable α- or γ-derivative of pyridine) with an amine derivative $H—N<^{R_1}_{R_2}$:



the addition of a reactive compound R—X (X = a halogen atom or a corresponding reactive negative group) to an α- or γ-aminopyridine:



the addition of a reactive compound R_2—X (for example a reactive alkyl or aralkyl halide or an acid halide) to an α- or γ-pyridonimide



For the application of the compounds as preserving and disinfecting agents the particular manner in which they are prepared is, of course, of no importance.

The active products in the form of their salts are in most cases colourless or faintly yellow, but rarely more deeply coloured compounds according to the manner in which they are made and their composition. They dissolve freely in water to solutions of nearly neutral reaction. Many members of the series also dissolve freely in organic solvents. Many of the substances have an appreciable capacity for promoting wetting, foaming and emulsifying, whereby their sphere of application is further enlarged.

The compounds can be used either in the chemically pure state or in the crude state. Furthermore they may be used in suitable admixture with one another or may be prepared from mixtures of parent materials. They may also be used in the form of mixtures or emulsions with other substances, which may be indifferent or may also be active.

According to the required solubility the compounds may be used in the form of their salts or in the form of the free bases. They have a very high bactericidal activity.

Examples of substances which may be used in accordance with the invention are the following:

1. N-Methyl-α-pyridone - stearoylimide hydrochloride.

2. N - Methyl - α - pyridone - cetylimide hydrochloride.

3. α - cetylbenzylamino - N - methyl-pyridinium bromide (obtained by reaction of 1 Mol. of N-methyl-α-pyridone-cetylimide with 1 Mol. of benzyl bromide).

4. α-Phenyl-stearoylamino - N - methyl-pyridinium chloride (obtained by the action of 1 Mol. of stearic acid chloride on 1 Mol. of N-methyl-α-pyridone-phenylimide; purified by dissolution in water and precipitation by dilute hydrochloric acid).

5. α-Naphthyl-nonoylamino-N-methyl-pyridinium chloride (obtained by the action of 1 Mol. of *n*-nonylic acid chloride on 1 Mol. of N-methyl-α-pyridone-naphthylimide, purified with ether).

6. N-cetylpyridinium chloride.

7. N-Cetyl-α-picolinium chloride.

8. Reaction product of cetyl chloride and γ-piperidinopyridine (apparently a γ-piperidino-N-cetylpyridinium chloride).

9. Reaction product of octadecyl chloride and γ-diethylaminopyridine (apparently a γ-diethylamino-N-octadecylpyridinium chloride).

10. Addition product of dimethyl sulphate and 3-stearoylaminopyridine (apparently 3-stearoylamino-N-methyl-pyridinium methosulphate).

The following may be given by way of illustration of the pronounced activity of the foregoing substances:

A suspension of Bacillus Coli is treated for ¼ hour with a dilute aqueous solution of the substance. It is then spread on an Agar plate and the growth of the Bacilli is observed. The Bacilli are killed at concentrations up to those shown in the following table:

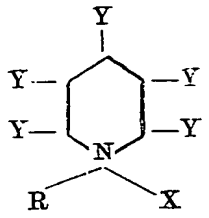
Compound No.	No growth at concentration of
1	1: 25000
2	1: 50000
3	1:100000
4	1: 5000
5	1: 10000
6	1:100000
7	1:100000
8	1:100000
9	1:100000
10	1: 25000

Instead of using the substances in aqueous solution they may be used in alcoholic solution or in solution in another suitable solvent.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be

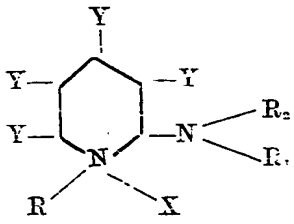
performed, we declare that what we claim is:—

1. A process for disinfecting and preserving, wherein there is used as disinfecting or preserving agent a quaternary pyridine of the general formula



in which X represents any desired anion or a hydroxyl group, Y represents hydrogen or any desired substituents which may be the same or different and R represents any desired organic residue.

2. A process as claimed in Claim 1, wherein there is used a derivative of an α- or γ-aminopyridine of the general formula



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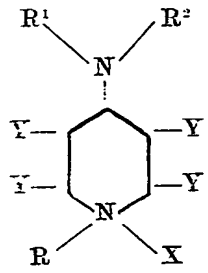
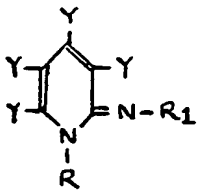


in which X, Y and R have the meanings indicated in Claim 1 and R_1 and R_2 represent hydrogen or the same or different organic residues.

3. A process as claimed in Claim 2 wherein in the general formula given in Claim 2 one or both of the residues R_1 and R_2 represents an acyl residue bound to the nitrogen in the manner of an amide.

4. A process of disinfecting or preserving, wherein there is used as disinfecting or preserving agent a pyridonimide of the formula



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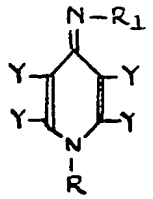

derived by the elimination of water or the acid H—X from a compound of the formula given in Claim 2 in which X, Y, R and R_1 have the meaning therein given and R_2 means hydrogen.

5. A process of disinfecting or preserving, wherein there is used as a disinfecting or preserving agent any of the compounds 1—10 herein named.

6. A manufacture of disinfectant preparations by mixing an agent defined in any of claims 1—5 with another such agent or with another substance.

7. A manufacture of disinfectant preparations by dissolving or dispersing in a liquid an agent defined in any of claims 1—5.

8. Disinfectant preparations obtainable by the manufacture claimed in claim 6 or 7.

Dated this 11th day of May, 1936.

ABEL & IMRAY.
Agents for the Applicants.

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